SOAKING TREATMENT OF BASSWOOD
At Low Temperatures

As affected by temperature and
viscosity of solution, wood moisture,
and type of treatment

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DEVELOPMENT OF THE "CLEAN TREATMENT" PROCESS, in which non-swelling, paintable (NSP) solutions of oil-soluble toxicants are commonly used, has helped to encourage wide use of the dipping and soaking methods of treating wood with preservative chemicals. These two methods of treatment are often used for farm, home, and industrial applications where pressure methods are neither practicable nor economically possible.

Current recommendations for the use of NSP solutions emphasize the desirability of treating only wood seasoned to moisture contents below 20 percent in solutions above 65° F. These recommendations are based on the results of a number of investigations, many of which involved pressure treatments. None of the investigations known to the author, however, were designed to study the interacting effects of the cold-soak treating variables under controlled conditions.

The scope of this investigation has been limited to the study of the cold-soak method and variables other than anatomical structure that are believed to have the greatest effect on solution absorption and penetration: solution temperature, wood moisture content at time of treatment, and type of treatment (intermittent or continuous). Although treating time is also an important variable (4)[1] it remained constant in this study.

Development and Present Status of the Cold-Soaking Method

A difference between soaking and dipping is commonly recognized. Dipping consists of submerging the wood in the preservative for a short time — usually less than 15 minutes — and then removing the product. Cold-soaking (hereafter referred to as soaking) consists of submerging the wood in a solution of an oil-soluble preservative for periods ranging up to several days at temperatures ranging from 65° to 80° F. (If the preservative is an aqueous solution, the process is called "steeping.")

Immersing wood in chemicals to improve its service life undoubtedly is one of the oldest methods of preservative treatment. Hunt and Gar-

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[1] Numbers in parentheses refer to literature citations on page 49.

ratt (18) mentioned Homberg's work with mercuric chloride in Europe in 1705. The soaking (or steeping) method probably was in use much earlier than the beginning of the 18th century, and has probably changed little since it was first used.

Earlier publications (17, 37, 38) recognized steeping, but the first recommendations for soaking did not appear in the literature until the late 1930's. Prior to the 1930's, the only commonly used oil preservative was creosote, including blends of it with petroleum and tar. The relatively high viscosity of these oils at room temperature tended to discourage interest in cold-soak treatments. Hubert (15, 16 — 1938) was probably the first to suggest the possibilities of applying pentachlorophenol in "light" oils by soaking and dipping.

In 1939 Carswell and Hatfield (7) reported on the merits of pentachlorophenol as a wood preservative, but they stressed the dip and pressure treatments and failed to mention the possibilities of soaking. Soaking was not used extensively until about 1940, when pentachlorophenol appeared on the market. Since 1940 a number of reports have been published on dipping or soaking wood (14, 15, 21, 26, 35, 40). Many of these investigators have studied the relation between wood moisture content and the absorption and penetration of preservative solutions.

Wohletz and Ravenscroft (40) reported that cold-soak treating results were best when the wood had a moisture content of 15 to 20 percent. They cautioned against treating fence posts at below-freezing temperatures, however, because they believed ice crystals that formed in the frozen wood stopped the treatment.

Moore and Ray (26) concluded that the length and character of the seasoning period for fence posts were more important in obtaining satisfactory results than the moisture content of the wood at time of treatment. They found that lodgepole pine posts seasoned below the fiber-saturation point (about 30 percent moisture) were not satisfactorily treated unless they had been seasoned for nine weeks or more.

Hicock and his colleagues (14) found that the best treating results were secured by thoroughly seasoning the wood and treating only during warm weather.

Marshall (21) concluded that the highly satisfactory results he obtained in his study of cold-soaking loblolly and shortleaf pine fence posts were due mainly to the method of seasoning. The retentions and penetrations obtained by this investigator decreased as seasoning time increased from 4 to 12 weeks and then increased as the time was increased to 16 weeks and longer. From these results, Marshall concluded

that glaze[1] or some other unknown factor inhibited treatment. Other investigators have also suggested the formation of glaze and surface hardening during seasoning as causes for unsatisfactory absorption and penetration of treating solution.

Most of the literature concerning the soaking treatment describes the use of warm preservative solutions. Some of the early recommendations on use of the soaking method were not supported by research, but they undoubtedly were based on pressure-treating recommendations (25). Bateman (2) concluded, after his pressure-treating study, that the viscosity of the treating oil was definitely related to the depth of penetration, and that the change in temperature (warming) of the oil had no other effect than to change the oil's viscosity. He observed that depth of preservative penetration increased as a result of reduced viscosity. MacLean (23), in his study of the effects of intermediate temperatures (160°-220° F) and viscosity of wood preservatives on penetration and absorption, found that during pressure treatment the viscosity of the preservative apparently had the most important single effect on penetration and absorption at any temperature, although the action of heat on the wood and the kind of oil used were also important. A year later (1927) MacLean (24) observed that the viscosity of creosote decreased with an increase in temperature, and the depth of penetration and absorption of the solution increased. Buckman *et al.* (6) reported similar results for the soaking method.

The only report known to the author of tests in which wood was treated with cold solutions was his study on winter treatment of white-pine fence posts published in 1948 (36). The results of treatment with solutions fluctuating in temperature between 14° and 26° F. were comparable to those obtained in a "warm" room (perhaps 75° to 80° F.). No attempt was made in this study, however, to control such important variables as wood moisture content or solution temperatures, to test solution temperatures below 14° F., or to study the interacting effects of solution temperature and wood moisture content.

There is a dearth of information on soaking wood in solutions below 65° F. for a number of reasons; among the most important are:

1. The poor absorption of solution and shallow depth of penetration usually obtained by soaking in comparison with pressure treatment.

2. The precipitation of some solutes when the solutions are cooled below a certain point.

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[1] Glaze was not identified, but it probably was the result of an accumulation of resins and other exudates on the surface during the early part of the seasoning period.

3. The relatively poor protection the cold-soak method often gives wood that is to serve in contact with soil.

In spite of these alleged shortcomings, however, there is a need for basic information on the cold-soak treatment, because the method has several desirable features. It is simple, economical, and practical. It requires only simple, inexpensive equipment, which is readily available. The sapwood of a few species can be treated as completely by soaking as by pressure methods. For these reasons a comprehensive investigation of the effects of solution temperature, wood moisture content, and type of treatment was initiated in order to determine the limitations of the method.

Procedures

EXPERIMENTAL MATERIALS

Pilot tests were made with ponderosa pine, but this species was abandoned because the treating solutions removed enough extractives to affect absorption weights. Basswood was chosen for the tests because a supply of sapwood was readily available; it has relatively low oil-soluble extractive content and a uniform texture; and it can be seasoned free of defects.

The test blocks for this study were obtained from one 10-foot bass-wood log about 17 inches in diameter. The log was selected from a number of freshly cut logs at a sawmill in Cass county, Illinois, for its good quality, uniform rate of growth, thick sapwood, and freedom from visible defects.

Boards 1¼ inches thick and 6 to 9 inches wide were removed "around the log" so that most of them were practically flat sawn and contained only sapwood. All boards were identified in such a way that their original position in the log could be determined. The green boards were end-coated at the mill with a commercial end-coating material and wrapped to prevent loss of moisture during transit.

In the laboratory the first eight boards removed from the outside of the log were judged to be free of heartwood and were then crosscut at mid-length. At the same time sections about 1 inch along the grain were removed from the center of each of three boards for moisture and specific gravity analyses. The 16 short boards were identified by number, weighed to the nearest 0.01 pound, end-coated with a commercial end-coating product, and stacked in a kiln for seasoning.

The three moisture-content samples were dried to constant weight at 221° F., and their moisture contents were calculated as a percent of oven-dry weight. The average moisture content, 97 percent, was used to calculate the oven-dry weight of each board.

The moisture-content samples also were used to calculate specific gravity. The average specific gravity of the three samples, based on oven-dry volume and oven-dry weight, was 0.48.

The "predicted conditioned weight" of each board for the particular level of wood moisture content at which treatment was to be made, namely, 7, 12, 19, or 26 percent, was determined from the calculated oven-dry weight, and the boards were weighed periodically until they reached these weights. The predicted conditioned weight (PCW) in pounds was calculated as follows:

$$PCW = COD + \frac{(COD)(DMC)}{100}$$

where COD was the calculated oven-dry weight in pounds and DMC was the desired moisture content in percent. The procedure was similar to that of using kiln sample boards to control the operation of a commercial dry kiln (11). The kiln schedule that was used (Table 1) was milder than the schedule recommended by the U. S. Forest Products Laboratory (34) for commercially seasoning basswood.

Table 1. — Kiln Schedule Used for Drying Basswood Lumber
for Test Blocks

Moisture content change (percent)		Dry-bulb temperature (° F.)	Wet-bulb depression (° F.)
From	To		
97	65	130	15
65	35	130	30
35	5	130	35

As the boards attained their predicted conditioned weights for each level of moisture content, they were removed from the kiln and surfaced on both sides to a thickness of 1 inch. Thickness was checked with a micrometer to the nearest 0.001 inch. The boards were then ripped into sticks 1¼ inches wide. The sticks were dressed until they were 1 × 1 inch in cross-section. The cross-sectional dimensions were checked with a micrometer to the nearest 0.001 inch.

Blocks. Four hundred eighty blocks 1 × 1 inch in cross-section and 6 inches long were cut, about 120 blocks each from boards of the same nominal moisture content. The blocks were cut to length with a planer saw. Variation in block length was controlled with a bar guide on the saw and a comparator gage measuring to the nearest 0.001 inch. Each block was inspected for freedom of defects, straightness of grain,

and smoothness of surface, and 400 were finally selected for treatment (100 of each nominal moisture content). Twelve of the rejected blocks (3 of each nominal moisture content) were subsequently used as moisture "check blocks" in the humidity chambers. Since longitudinal penetration (along the grain) is greater — as much as 75 times for some species (4) — than penetration across the grain, the ends of each block were given two coats of a phenolic resin adhesive, applied 24 hours apart, to prevent end penetration by the treating solutions.

Test wafers. Two types of test wafers $1 \times 1 \times \frac{1}{4}$ inch along the grain were used in the investigation: a penetration wafer for measuring toxicant content at various depths in the test blocks and a moisture-solvent-interchange wafer. The preparation and use of these wafers are described on pages 17 and 20.

Toxicant. Triple recrystallized pentachlorophenol was used to prepare the treating solutions. The purity of the chemical (99.9 percent) was at least the equivalent of an analytical reagent grade.

Solvents. Diacetone alcohol (4-hydroxy-4-methyl-2-pentanone) and hexylene glycol (2-methyl-2,4-pentanediol) were chosen from a number of organic compounds that had the properties set as standards for the solvents. Both of these solvents had low vapor pressures and high solubility for pentachlorophenol at temperatures as low as 5° F. Because they had different viscosities, the diacetone alcohol was classified as a "light" solvent and the hexylene glycol as a "heavy" solvent. The properties and characteristics of the two compounds are shown in Table 2.

The solvents were not reagent grade, although they met rigid manufacturing specifications. They were essentially anhydrous when manufactured, but they could have contained as much as 0.2 percent[1] of water by weight by the time they were used.

Treating solutions. The pentachlorophenol was dissolved in each of the solvents at the rate of 5 gm. per 100 cc. of solvent. Ordinarily pentachlorophenol solutions contain toxicant at a concentration of 5 percent of the solution weight; however, in this investigation differences in solution absorption and penetration were used to show the effects of treatment. Thus it was important that each unit volume of solution contain the same amount of toxicant regardless of the specific gravity of the solvent. Both solutions were analyzed for toxicant by the lime-ignition method (8) and the concentration of pentachlorophenol

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[1] Information contained in letter to author from C. F. Brice, Shell Chemical Corporation, dated November 3, 1955.

Table 2. — Properties of Test Solvents for Pentachlorophenol

Property	Diacetone alcohol (4-hydroxy-4-methyl-2-pentanone)	Hexylene glycol (2-methyl-2, 4-pentanediol)
Molecular weight............................	116.16	118.17
Specific gravity, 20°/4° C....................	.9382	.9216
Boiling point, ° C. at 760 mm. Hg.............	169.1	198.3
Freezing point, ° C...........................	−44	...[a]
Flash point, ° F.............................	144[b]	210[c]
Vapor pressure, mm. Hg. at 20° C.............	.81	.05
Viscosity, centistokes		
Cold condition...........................	15.3[d]	700.0[e]
Warm condition..........................	...[f]	32[g]
Weight, lb. per gal. at 20° C.................	7.83	7.69
Solubility for pentachlorophenol at 5° F., percent		
by weight................................	31	32
Type of solvent...........................	Light	Heavy

[a] Becomes semi-solid at −40° C. without crystal formation.
[b] Tag. Open Cup.
[c] Cleveland Open Cup.
[d] −15° C. (5° F.).
[e] At approximately 10° F.
[f] Data not available.
[g] At approximately 80° F.

balanced until Student's "t" test showed no significant differences be-tween the mean concentrations of the aliquot samples.

Reagents. All reagents used in analyzing the wood samples for pentachlorophenol were analytical reagent grade and met the specifica-tions adopted by the American Wood-Preservers' Association in their Standard Method A5-54 (8).

DESIGN OF EXPERIMENT

The design of this experiment was $5 \times 4 \times 2 \times 2$ factorial relat-ing the effect of solution temperature, wood moisture content, solvent viscosity, and type of soaking treatment to preservative absorption and penetration. The 80 five-block groups were randomly assigned to treat-ments. Four groups formed a run. Each run was treated at one of the five levels of solution temperature, and all tests blocks in the run were of the same moisture content. No account was taken in the statistical analyses of the deviation of block moisture contents from the nominal values, although measured values were used in plotting the graphs.

The results will be discussed in three sections. One section concerns wood moisture-solvent interchange. Another section concerns the ef-fects of the various treating variables on the absorption of preservative solution by the test blocks, and the final section concerns the effects of the variables on the concentration of toxicant at three mid-point depths in the blocks.

Constant-humidity chambers were used to condition test blocks to controlled moisture contents. Each chamber held about 125 blocks. The parts are: (1) floating bearing block, which supported stirring-rod shaft; (2) container for saturated salt solution, which also supported expanded metal shelf on which blocks were stacked for conditioning; (3) stirring rod (note propeller blade, which circulated air in chamber); (4) test blocks stacked for seasoning (note small stickers separating each course of blocks); and (5) chamber lid (note two observation ports, which were sealed with clear plastic sheeting, and rod port, which was covered with sheet rubber gasket). Motor that powered stirring rod is not shown.   (Fig. 1)

Table 3. — Environment and Saturated Salt Solutions Used to Obtain
Controlled Moisture Contents of Basswood Test Blocks

Wood moisture content (percent)		Environment used to obtain moisture content		Saturated salt solution
Nominal	Actual	Temperature (° F.)	Relative humidity (percent)	
5	7	75	20	$KC_2H_3O_2$
12	13	75	66	$NaNO_2$
19	20	75	88	K_2CrO_4
26	28	41	95	$ZnSO_4 . 7H_2O$

EQUIPMENT

Constant-humidity chambers. The blocks were stored in closed constant-humidity chambers (Fig. 1) over saturated salt solutions to obtain four levels of nominal moisture content: 5, 12, 19, and 26 percent. Table 3 shows the approximate environment and the salt solutions used to obtain the controlled moisture contents. The solutions of chemically pure salts contained an excess of solute so that a constant water-vapor pressure (relative humidity) would be maintained above their surfaces at a reasonably constant temperature. The chamber containing zinc sulfate was stored in a refrigerator at 41° F. to prevent mold and stain fungi from developing on the blocks during conditioning.

An electrically driven fan constantly circulated the air in each chamber, and a stirring device circulated the saturated salt solutions.

Constant-temperature chamber. Figs. 2 and 3 show the constant-
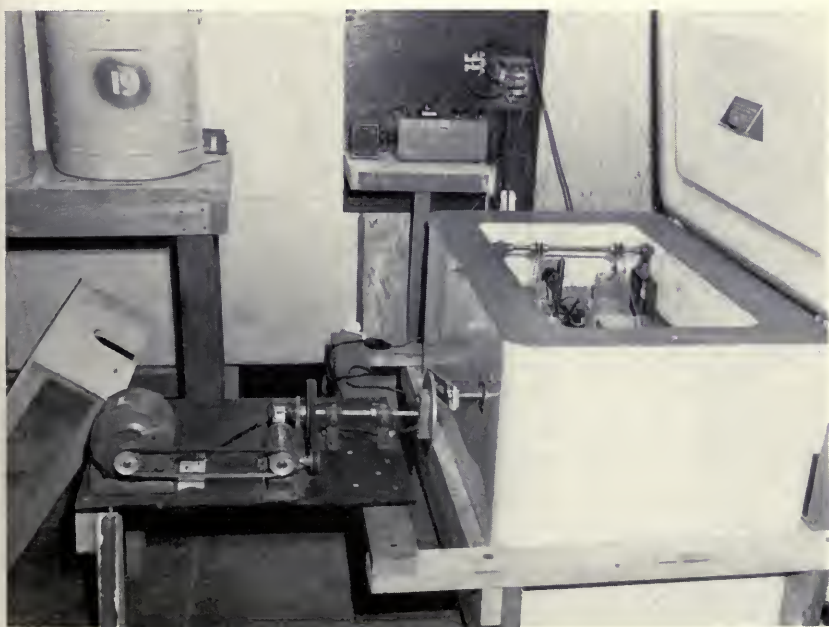
Constant-temperature chamber for treating test blocks. Electric motor shown in lower-left corner powered the intermittent dipping system. To the right of the motor is the speed reducer, and immediately behind it is the counting device that tallied the number of dips given blocks in the treating baskets. The potentiometer on the small shelf in the background was used to measure solution temperature in the treating tanks. Both heating and cooling units were operated through the relay system shown on the instrument panel. (Fig. 2)

temperature chamber used to control solution temperatures during treatment. A food freezer was modified and equipped with a water-bath tank and temperature-regulating devices so that the blocks could be treated at various levels of solution temperature between 5° and 80° F. Adding a commercial antifreeze solution to the water prevented it from freezing, and an electric motor-driven stirrer constantly circulated the water. Four 4 × 5 inch sheet-steel treating tanks about 12 inches deep were set 9 inches deep in the water bath.

The temperature of the water bath was controlled by a mercury thermoregulator that operated a 250-watt strip heater and the food
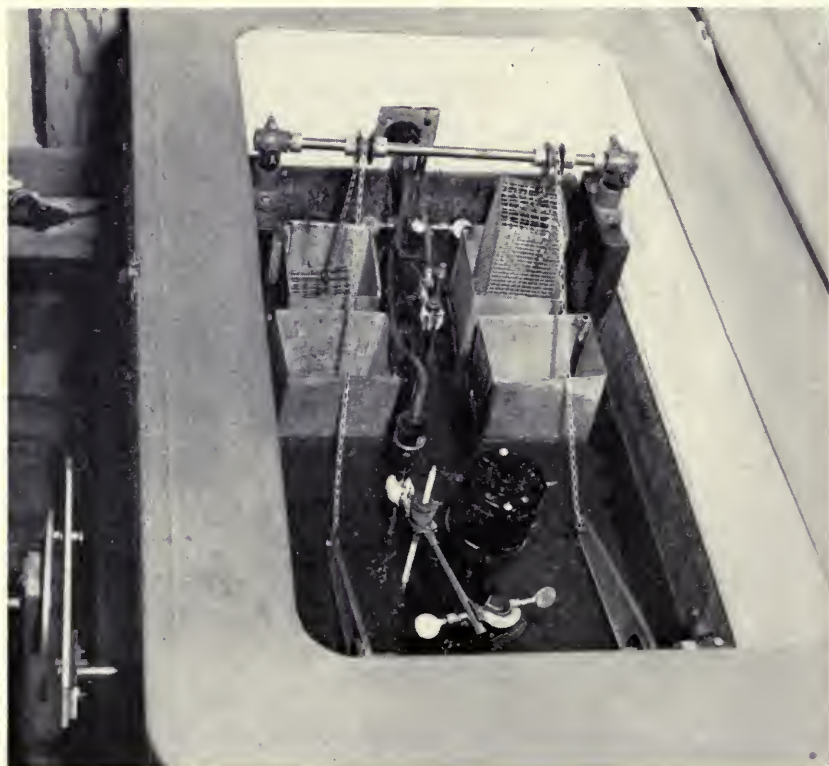
Treating tanks. Shown from front to rear: (1) two plastic pitman arms that activated the dipping baskets by chains; (2) to the right of the mercury thermometer, the electric stirrer that circulated the water-bath solution; (3) a 250-watt strip heater to the rear of the thermometer; (4) the two tanks in which blocks were continuously treated — these had no wire baskets; (5) a single-tube mercury thermoregulator between the treating tanks; (6) the small vertical tube in one corner of each treating tank which served as a holder for a copper-constantan thermocouple.   (Fig. 3)

freezer's compressor through a sensitive electronic relay. The thermo-regulator was sensitive to ±0.1° F. temperature change.

Copper-constantan thermocouples were located in each treating tank; however, only one thermocouple was used during treatment be-cause potentiometer readings made during the pilot tests showed that the range in solution temperatures was less than 0.1° F.

A 75:1 speed reducer and a pulley-belt arrangement reduced the speed of a ⅓ HP electric motor to about 7 rpm. Fig. 3 shows the system of mechanical levers, eccentric, shafting, and pulleys that dipped two baskets into tanks of treating solutions at the rate of about seven times a minute, or 5,100 times in 12 hours, the length of all treatments. A counter automatically measured the number of dips given the blocks during intermittent treatment.

OBTAINING ABSORPTION AND PENETRATION DATA

It was difficult to obtain satisfactory moisture conditions in the 26-percent-moisture group. Fungal growth developed on one set of test blocks in a humidity chamber at room temperature (75° F.), and a substitute group of blocks was prepared from material that had been restored to green condition in distilled water after seasoning to about 24-percent moisture. The reconditioned blocks, containing about 45-percent moisture, were then stored in a humidity chamber at 41° F. until they were removed for treatment at 28-percent moisture.

Blocks were stacked in spaced layers with ¼-inch-square stickers separating each course (Fig. 1). The arrangement of blocks in the chamber permitted air to circulate freely among them. As a result of the humidity maintained in each chamber, the blocks attained an equilib-rium moisture content in two to six weeks. Periodic checks were made with an electrical resistance-type moisture meter on three "check blocks," which were placed in each chamber along with the test blocks, and on the samples cut from the check blocks and oven-dried to con-stant weight at 221° F. The test blocks were treated as soon as the measurements showed that they had reached the proper moisture content.

Twenty test blocks were removed from a humidity chamber and stored in a polyethylene bag — except for a short time when each block was being numbered, measured, and weighed — until they were placed in the treating tanks at room temperature.[1]

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[1] No tests were made to check the rate at which temperature of the blocks reached equilibrium with temperature of the solution; however, Boller (3) re-ported that blocks of Douglas fir of similar size were cooled from room tem-perature to —300° F. in about 12 minutes.

A jig (Fig. 4) was used to mark the location of the penetration wafer and the point at which the test block was numbered. Thus the number remained on the wafer when it was sawn from the block following treatment.

Ten blocks were measured before and after they had been end-coated with adhesive, and the average thickness of the end-coatings was found to be 0.048 inch. This amount was subtracted from the gross length of each block before its cubic content was calculated.
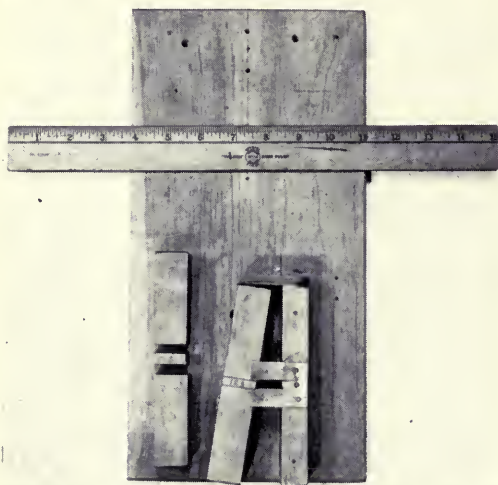
Jig for marking location of penetration wafers on test blocks. After block was marked, it was numbered; this number also identified the penetration wafer. Block at left has been crosscut to remove a wafer ¼ inch along the grain.   (Fig. 4)

Dimensional measurements were made on one block selected at random from the group stored in the 26-percent conditioning chamber to determine whether the conditioned blocks were below the fiber-saturation point. The three structural dimensions of the block were measured to the nearest 0.001 inch, the block was returned to green condition in distilled water by a vacuum treatment, and the dimensions were remeasured. The dimensions of the block increased by the following percentages: tangential, 4; radial, 2; and longitudinal, less than 0.01. Although measurements were limited to one block, the swelling indicated that the blocks represented by the random sample were treated at slightly below the fiber-saturation point.

Each block was weighed to the nearest 0.01 gm. before and after it was treated.

Each group of five blocks was placed in a wire basket for treatment.

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Two five-block groups received continuous treatment for 12 hours,[1] one in the diacetone alcohol solution of pentachlorophenol and the other in the hexylene glycol solution. Two other groups were dipped 5,100 times in the 12-hour treating period, one group in each of the two solu- tions. Although all blocks were treated 12 hours, those treated by inter- mittent dipping were not constantly submerged in the treating solution during this period. In order to determine the percent of time the blocks were actually submerged in the solution, the length of time the blocks were completely submerged was measured with a stopwatch during a 7-minute sample treating period. Submerged time was calculated as a percent of total time. It was assumed that the proportion of the time the blocks were submerged during the sample period was the same as that for the 12-hour treating period.

The treated blocks were wiped dry before they were reweighed. However, although the blocks were weighed promptly following treat- ment, some bleeding[2] was observed. Therefore, each block was wiped free of solution once again before it was crosscut at mid-point, and a wafer $\frac{1}{4}$ inch along the grain removed to analyze penetration.

Each wafer was cut into three parts (Fig. 5): Shell A, the outer $\frac{1}{8}$-inch layer; Shell B, the second deepest layer, also $\frac{1}{8}$ inch thick; and the core, the remaining innermost portion of the wafer. The wafers were cut into shells and cores within one hour after the blocks were removed from the treating tanks to minimize "creep" of the solution following treatment.

The penetration analysis was simplified by grouping shells and cores. For example, all of the A shells cut from each group of five similarly treated blocks were ground together in a burr mill. The B shells and cores were similarly homogenized. Thus the 15 depth-of-penetration samples cut from each group of five blocks were reduced to three "average" samples. The grinding process reduced the samples to the fineness of sawdust, 97 percent of which passed a No. 10 screen (100 openings per square inch). About 53 percent of the sample was re- tained by a No. 20 screen; 32 percent was retained by a No. 40 screen; and 12 percent passed a No. 40 screen. The burr mill was thoroughly cleaned with ethyl alcohol between grindings.

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[1] Pilot tests showed that the treating solutions penetrated the blocks a "maximum allowable depth" in about 12 hours. The maximum allowable depth of penetration permitted treatment of a block's cross-section to a point just short of complete saturation; thus the core received little, if any, treatment. Soaking periods longer than 12 hours often resulted in complete saturation of a few blocks; complete saturation obscured the effects of treatment.

[2] Bleeding is the exudation of solution on the surface of the wood.

Jig for cutting shells and cores from penetration wafers. Wafer in jig shows location of three samples analyzed for toxicant content. At left is the group of shells and cores (three samples from each of five blocks) for one of the 80 treatments. The pile of "sawdust" shows how each group of shells or cores was ground to a single, homogenized sample by a burr mill.   (Fig. 5)

A sample of ground wood from each shell and core of each group was analyzed by the lime-ignition method adopted by the American Wood-Preservers' Association (8) as the standard method for calculating the percent of pentachlorophenol in wood. The percent of pentachlorophenol was determined on a weight basis for two reasons: (1) The moisture content of the ground wood was considered a constant following four months' storage, and (2) it was more accurate to weigh the sample than to determine its volume.

Fig. 6 shows the mark-sense card on which thickness, width, length, and untreated and treated weights were recorded. The concentrations of pentachlorophenol found in Shells A and B and the core were recorded on the back of the card. With minor exceptions, all data were organized and computed by business machines from the measurements coded on the mark-sense card. Cubic-inch volumes were converted to cubic centimeters by a conversion factor. Where absorption of solu-

tion has been shown in pounds per cubic foot, the values were obtained by multiplying metric data (gm./cc.) by 62.43.

One 5-cc. and one-10 cc. sample of each treating solution were dried to constant weight at 80° to 85° F. three and a half months after the treatments were completed, and the amount of extractives removed from the basswood was calculated as a percentage of weight of solvent and extractive materials.
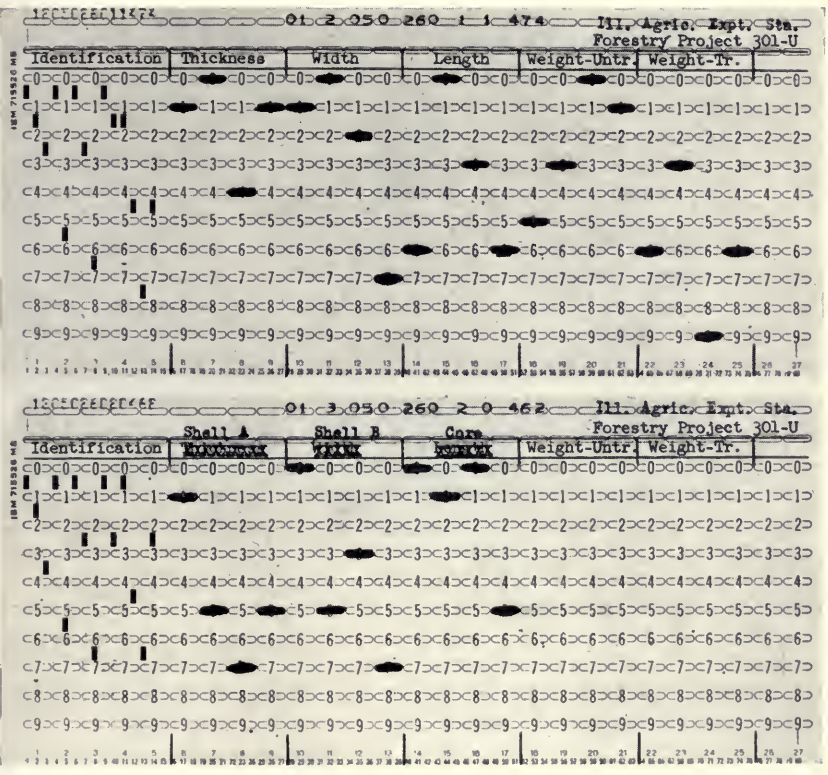
Mark-sense cards used to record size, weight, and toxicant-concentration data. Identification data in the first 11 columns were interpreted near the top edge of the card for ease in reading. For example, the top card shows size and weight data for Block 474. Reading from left to right, block was in treating run 01, group 2, and was treated in solution having a temperature of 05.0° F. at 26.0-percent moisture content. The solution was diacetone alcohol (coded "1") and the intermittent (coded "1") soaking method was used. The remaining numbers are the block numbers. The mark-sense codings were subsequently punched into each card. The bottom card is from a second deck, modified to record penetration data. The percent of pentachlorophenol found by laboratory analysis (1.575) was recorded in mark-sense position 6 to 9 with a special graphite pencil.   (Fig. 6)

OBTAINING MOISTURE-SOLVENT INTERCHANGE DATA

A test was made to determine whether there was an interchange of solvent and the moisture in the wood during treatment. This test was divided into two phases.

The objective of the first phase was to determine whether the two solvents, diacetone alcohol and hexylene glycol, penetrated the cell walls during treatment. Hawley (12), DeBruyne (9), Stamm (29), Nayer (27), and a number of other investigators have shown that the wood swells if water or other liquids penetrate the capillary structure of the cell wall. A number of theories have been given to explain why the wood swells (30), but all that is of interest here is that swelling or dimensional changes do occur, and these changes in dimensions indicate cell-wall penetration.

The objective of the second phase of the test was to determine whether the solvents absorbed moisture from the wood.

A third possibility also existed, namely, that water absorbed from the wood would be replaced by solvent.

Fifty-two wafers 1×1 inch in cross-section and $\frac{1}{4}$ inch along the grain were randomly selected from a group of 60 cut from two green basswood[1] squares about 15 inches long. The squares were ripped from a single board and dressed to size before 30 wafers were cut from each of them. The wafers were consecutively cut from the squares with a smooth-cutting planer saw; the $\frac{1}{4}$-inch longitudinal dimension of each wafer was controlled by a spacing jig on the table saw. The annual rings in all wafers were practically parallel to the tangential plane of growth.

The wafers were numbered consecutively and assigned randomly to treatments.

Thirteen wafers were exposed in each of four humidity chambers over saturated salt solutions (Fig. 7). The radial and tangential dimensions of each wafer were measured periodically to the nearest 0.001 inch (Fig. 8) until they reached a constant size after 93 hours of exposure. The salt solutions in the humidity chambers were selected to provide four levels of controlled equilibrium moisture content, namely, 5, 12, 19, and 26 percent (Table 4).

At the end of the moisture-conditioning period, five wafers having a common moisture content were measured radially and tangentially and submerged in an 8-ounce, wide-mouthed jar containing one of the

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[1] Average moisture content of three samples of the freshly sawn basswood was 95 percent, based on oven-dry weight. Average specific gravity of the three wood samples was 0.37, based on oven-dry weight and oven-dry volume.

Constant-humidity chambers for conditioning test wafers in moisture-solvent interchange study. Bottom well contained a saturated salt solution that was stirred constantly by motor-driven stirring rods. Each stirring-rod shaft also turned a propeller blade that circulated the air inside the chamber. Jars of solvent are shown (front chamber on right) in the chambers; however, tests showed that it was impractical and unnecessary to condition the solvents, and the practice was discontinued. Test wafers were conditioned in a basket of hardware cloth supported on a hardware-cloth standard to prevent possible contamination by the salt solution.   (Fig. 7)

solvents. One 5-wafer group of each nominal moisture content was treated in each of the two solvents.

The three untreated wafers remaining in each humidity chamber were weighed, dried to constant weight at 221° F., reweighed, and their average moisture content calculated as a percent of their oven-dry weight. The treated wafers were assumed to have the same moisture content as those analyzed for moisture content.

Table 4. — Environment and Saturated Salt Solutions Used to Obtain
Controlled Moisture Contents of Basswood Test Wafers

| Wood moisture content (percent) | | Environment used to obtain moisture content | | Saturated salt solution |
Nominal	Actual	Temperature (° F.)	Relative humidity (percent)	
5	7	70	32	$CaCl_2 . 2H_2O$
12	10	70	52	$Na_2Cr_2O_7$
19	16	70	81	$(NH_4)_2SO_4$
26	26	43	95	$ZnSO_4 . 7H_2O$

The jars containing the solvents and blocks were quickly capped and stored for 12 hours in a water bath at 80° F. At the same time that the wafers were placed in the solvents for treatment, a sample of each solvent was similarly stored for subsequent moisture analyses. All jars were equipped with paper and aluminum-foil cap-liners to prevent the solvents from gaining or losing moisture to the atmosphere.

At the end of the treating period the wafers were removed from the jars, wiped dry, and remeasured. The moisture content of each of the

This comparator gage was used to measure basswood wafers. Each wafer was indexed for measurement by moving it to the rear and to the left side of the slot. To calibrate the gage with a 1-inch standard end measuring rod, a plastic filler strip was inserted in the slot to make gage readings fall between 1 and 2 inches. The thickness of the filler strip, 0.244 inch, was later subtracted from each measurement.   (Fig. 8)

10 samples of solvents was determined by the Karl Fischer method (1), using commercially prepared reagent and alcohol-water solutions. The end-point of each titration was determined electrometrically. Duplicate tests were made, but the analyses showed that the second measurements were consistently higher than the first. Since exposure of the solvents to the atmosphere apparently had some effect on the results, the original measurements were used and the duplicate tests abandoned.

Results and Discussion

No difficulty was experienced in bringing the blocks in the 5-, 12-, and 19-percent moisture groups to a uniform moisture content at room temperature, although actual values exceeded nominal percentages by not more than 2 percentage points. The 26-percent group, however, required a temperature lower than those prevailing in the laboratory to prevent the development of fungi during the final conditioning treatment. Moisture content variations within a group of blocks were within a range of 2 percentage points.

No complete or continuous records were obtained of the variation in solution temperatures. However, the equipment was sensitive to changes of $0.5°$ F. and the variations recorded were less than $+2°$ F. The consistent overrun of temperatures was attributed to heat from the electric motor on the stirrer.

Dimensional changes which developed after the blocks were end-coated and placed in the humidity chamber for conditioning apparently caused very fine cracks (crazing) to develop in the end-coatings on blocks in the 26-percent-moisture group. How much solution penetrated through the small cracks and along the grain is unknown. However, 10 treated blocks were crosscut at various distances from the ends and examined for evidence of solution penetration. None of the blocks examined showed solution penetrations deeper than 1 inch along the grain. Those blocks that were penetrated through the end-coating showed only an anastomosing network of fine lines corresponding to the pattern of cracks. The question, to what extent did longitudinal penetration affect liquid absorption in blocks of the 26-percent-moisture group, may be asked. Although no data are available on which an answer can be based, the effect is believed to be insignificant. If the longitudinal penetration did significantly increase absorption, the amount of change probably would be constant for all variables except moisture content.

Constant agitation of the hexylene glycol solution during intermittent treatment caused the liquid to froth at temperatures below 40° F. No froth was observed in the diacetone alcohol solution at any temperature.

Bleeding of solution occurred on blocks treated at solution temperatures below 40° F. The exudation probably resulted from the increase in temperature and expansion of air or liquid, or both, that took place in the blocks during the final weighing and sawing of penetration wafers. For example, blocks treated at 10° F. and 12-percent moisture content were weighed immediately after treatment (as were all blocks) and again 30 minutes later. The loss in weight from bleeding was about 0.5 percent. There was no difference in loss of weight over that period between intermittent and constant treatments, but the weight loss for blocks treated in the hexylene glycol solution was 2.4 times that for those treated in the diacetone alcohol solution.

Some smearing of the solutions across the cross-section occurred during the sawing of the penetration wafers. Based upon color differences, the smearing appeared to be limited to the outer ¼-inch portion of the wafer. There was no practical means of controlling the smearing. It is believed, however, that the degree of contamination of shells and cores during sawing was constant for all treatments, and that contamination did not affect the significance of the results.

The color of both treating solutions had darkened considerably by the time all blocks were treated, indicating that some extractive materials had been removed during the treating process. An analysis of two samples of each solution showed that 1.63 percent of the weight of the diacetone alcohol solution and 1.36 percent of the weight of the hexylene glycol solution were extractives. The identification of the extractive materials was not within the scope of this investigation.

Many factors controlled the movement of the treating solutions through the test blocks, but only solution temperature, wood moisture content, and type of treatment and solution were controlled. The extent to which the controlled factors affected solution absorption and penetration depended largely on the types of anatomical elements penetrated by the solutions. Thus before evaluating the absorption and penetration data, it is important to know whether the cell-wall capillaries were penetrated by the treating solutions. This was the main reason for measuring the moisture-solvent interchange.

WOOD MOISTURE-SOLVENT INTERCHANGE

Tables 5, 6, and 8 and Figs. 9, 10, and 11 show the results of tests to determine whether there was an interchange of moisture in the wood

and diacetone alcohol or hexylene glycol during treatment. Table 7 shows the analysis of variance employed in testing the significance of treating effects on radial and tangential dimensions. The error correlation between the two dimensions was tested and found not significant; thus the effects of treatment on each dimension will be examined separately.

The potential effects of the soaking treatment were these:

1. Solvent could have entered the capillary structure of the cell walls and caused swelling.

2. Hygroscopic moisture could have left the capillary structure of the cell walls and caused shrinkage.

3. A combination of the two previously listed possibilities could have occurred. In this event, all degrees of liquid interchange could have taken place, and the results would have been disclosed as dimensional changes in the wafers and as an increase in the moisture content of the solvents as a result of treatment.

Table 5 shows that the average radial dimensions of the wafers shrank upon treatment at all wood moisture contents below 26 percent; treatment at 26 percent moisture content resulted in swelling. The average radial dimension was 0.986 inch before soaking in the organic solvents and 0.984 after treatment (Table 6). The amount of shrinkage measured is small, but the moisture content of the wood had a highly significant effect on the radial dimensional changes and so did solvent (Table 7). Changing solvents had the same effect on the radial dimen-

Table 5. — Average Dimensions of Basswood Wafers With Different Moisture Contents Before and After Soaking in Diacetone Alcohol and Hexylene Glycol for 12 Hours at 80° F.

Wood moisture content (percent)	Average dimension (inches)			
	Radial		Tangential	
	Before soaking	After soaking	Before soaking	After soaking
Treated with diacetone alcohol				
7	.974	.973	.922	.929
10	.982	.977	.933	.940
16	.989	.987	.942	.947
26	1.000	1.004	.955	.965
Treated with hexylene glycol				
7	.973	.970	.921	.919
10	.983	.975	.932	.928
16	.990	.987	.941	.940
26	1.001	1.002	.960	.968
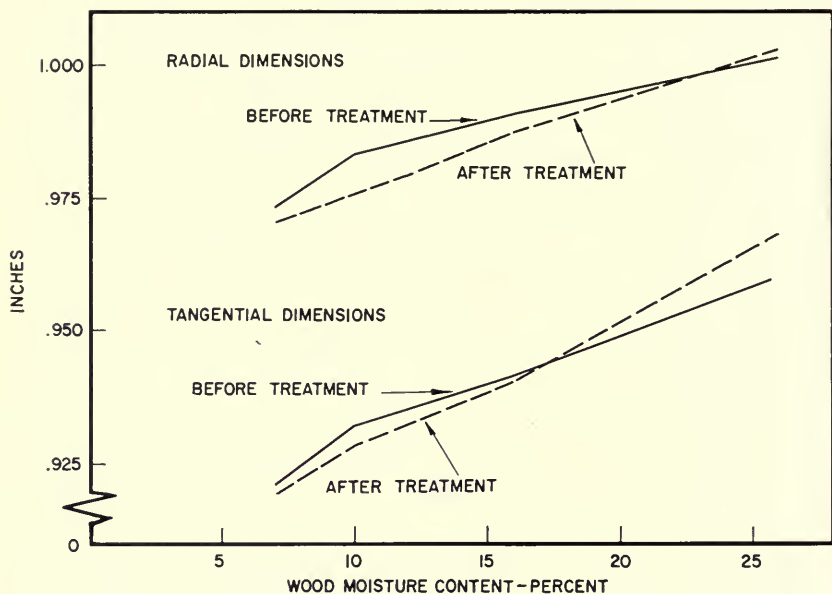
Effect of a 12-hour soak in hexylene glycol on the dimensions of basswood
wafers treated at different moisture contents.   (Fig. 9)
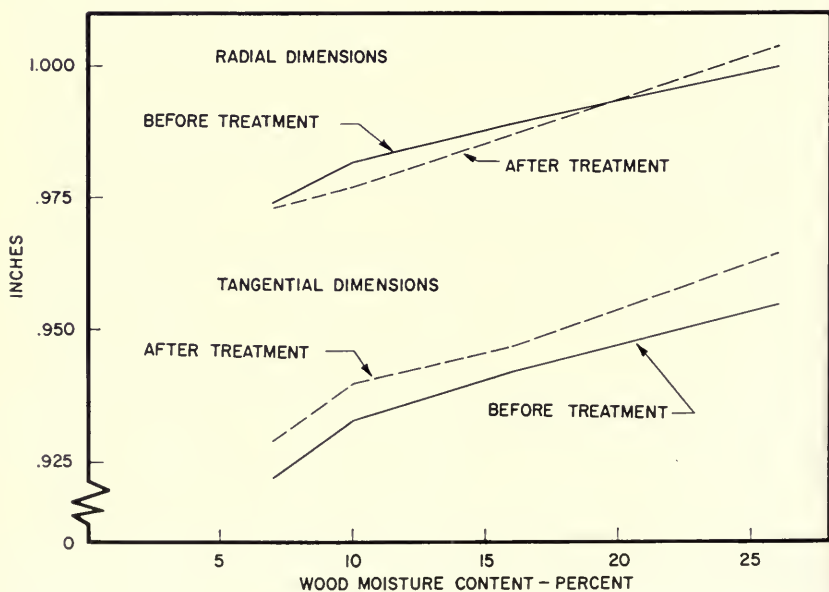
Effect of a 12-hour soak in diacetone alcohol on the dimensions of bass-
wood wafers treated at different moisture contents.   (Fig. 10)

Table 6. — Dimensional Change in Basswood Wafers of Different
Moisture Contents Before and After Soaking 12 Hours
in Organic Solvents at 80° F.

Wood moisture content (percent)	Average dimension (inches)				Dimensional change (percent)[a]	
	Radial		Tangential			
	Before soaking	After soaking	Before soaking	After soaking	Radial	Tangential
7	.973[b]	.972	.921	.924	−.103	.326
10	.982	.976	.932	.934	−.611	.215
16	.989	.987	.942	.943	−.202	.106
26	1.000	1.003	.957	.966	.300	.940
Average	.986	.984	.938	.942	−.203	.426

[a] Dimensional change expressed as percent of untreated dimension.
[b] Each average based on a measurement of 10 wafers, half of which were soaked in diacetone alcohol and the remainder in hexylene glycol.

sion at each level of moisture content as shown by the nonsignificant
MS interaction in Table 7.

In studies of the dimensional changes in wet wood following heat-
ing, Koehler (19) and Wise and Jahn (39) have attributed the de-
crease in the radial dimension to crooking of the rays or corrugation of
the ray cells as a result of uneven tangential swelling. The crooking
probably was caused by greater tangential than radial stresses which
developed during the treating process.[1]

The average tangential dimensions at each level of wood moisture
content increased as a result of treatment (Table 5). The average
tangential dimension of all wafers tested at all levels of wood moisture
content swelled from 0.938 inch to 0.942 inch upon treatment (Table
6). The amount of swelling is small, but highly significant. Table 7
shows not only that the moisture content of the wood at time of treat-
ment and solvent had significant effects upon the tangential dimensions,
but also that the MS, moisture by solvent, interaction was significant at
the 0.001 level of probability. Thus a change in solvents produced
different effects on the tangential dimension at different levels of wood
moisture content. The largest amount of swelling occurred in the wa-
fers treated at 26-percent moisture content. The greatest swelling at
the highest moisture content is attributed to the expansion of the cell-
wall capillaries which occurred with an increase in the moisture content
of the wood.

[1] No check was made to determine whether stresses were present in the
wafers following the conditioning or treating processes; however, if stresses
were present, the strain was not disclosed as a change in the square form of
the wafers.

Table 7. — Analysis of Variance for Wood-Moisture-
Solvent Interchange Data

Source of variation	Degrees of freedom	Radial dimensional change[a] (x)			Tangential dimensional change[a] (y)		
		Corrected sum of squares	Mean square	Variance ratio (F)[b]	Corrected sum of squares	Mean square	Variance ratio (F)[b]
Moisture content of wood at time of treatment (M)	3	413.9750	137.9916	28.5***	376.0750	125.3582	22.5***
Solvent (S)	1	46.2250	46.2250	9.6**	540.2250	540.2250	97.1***
Moisture × solvent (MS)	3	9.9750	3.3250	.7 NS	120.4750	40.1583	7.2***
Error	32	154.8000	4.8375	....	178.0000	5.5625	....
Total	39	624.9750	....	....	1214.7750	....	....

[a] Each measurement of dimensional change multiplied by 1,000.
[b] *** Significant at .001 level of probability.
** Significant at .01 level of probability.
NS, not significant.

There is also the possibility that the size of the solvent molecule
prevented it from readily penetrating the cell-wall capillaries at the
lower moisture contents. Nayer (27) found that, as the size of the
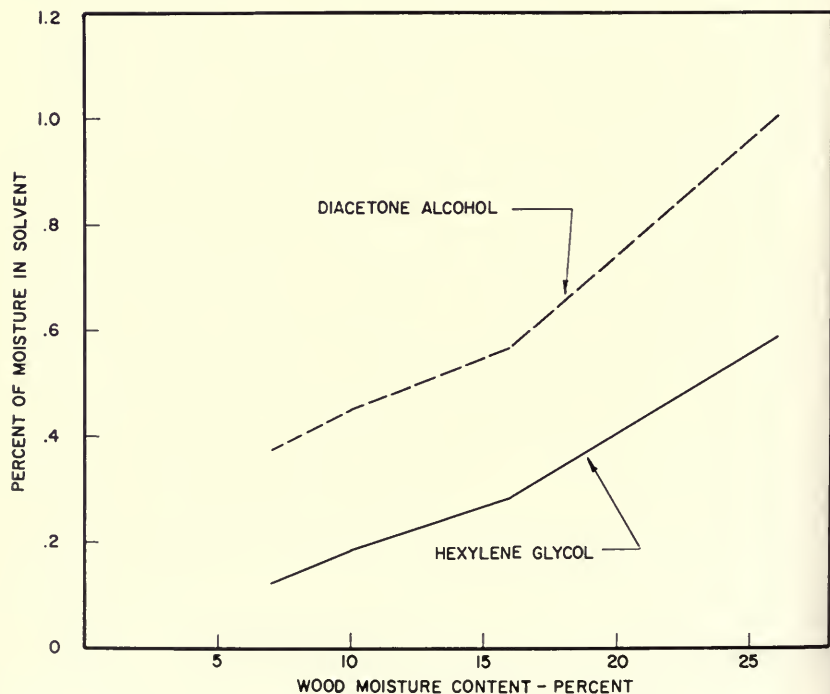
Percent of moisture in solvents following treatment of basswood wafers of
different moisture contents.   (Fig. 11)

Table 8. — **Moisture Content of Diacetone Alcohol and Hexylene
Glycol Before and After Soaking Basswood Wafers
of Different Moisture Contents**

Wood moisture content (percent)	Moisture content of solvent (percent)		
	Diacetone alcohol	Hexylene glycol	Average
Before soaking			
....	.191	.048	.119
After soaking			
7..	.377 (197)[a]	.123 (256)	.250 (210)
10...	.451 (236)	.185 (385)	.318 (267)
16...	.568 (297)	.288 (600)	.428 (360)
26...	1.003 (525)	.591 (1231)	.797 (670)
Average..................................	.600 (314)	.297 (618)	....

[a] Numbers in parentheses show final moisture contents of solvents expressed as percent of their original moisture contents.

molecule increased, there was an increase in the width of the molecules (due to thermal effects) and in resistance to diffusion.

Nayer also reported that Hermans (13) found anhydrous glycerine did not diffuse into dry cellulose, but when a small amount of water was added to the glycerine, the water was selectively adsorbed by the cellulose and the resulting swelling created capillaries through which glycerine diffused. Thus at 28-percent moisture content the cell-wall capillaries would be swollen to maximum size and would permit molecules to enter that would be unable to penetrate at lower moistures.

The moisture contents of the solvents in which the wafers were soaked were measured before and after the wafers were treated. Table 8 and Fig. 11 show the results of the measurements. At each level of wood moisture content, some of the moisture in the wood was absorbed by the diacetone alcohol and hexylene glycol. The amount of moisture absorbed by the solvents increased positively with an increase in wood moisture content. The diacetone alcohol absorbed a greater amount of moisture from the wood than the hexylene glycol. The compound is completely miscible with water, and it is used commercially as a solvent. The difference in moisture absorption, however, is attributed to the better penetrating characteristics of diacetone alcohol.

Thus it appears that some moisture in the wood was absorbed by

the solvents and that some of the solvents penetrated the cell-wall capillaries during treatment. The extent of the liquid interchange depended upon the amount of moisture in the cell walls at the time of treatment and the kind of solvent used to treat the wood.

This means that the absorption figures given subsequently are probably conservative, since the moisture lost from the wood during the 12-hour soaking period was replaced by treating solution. The moisture absorbed by the treating solution, however, reduced the concentration of toxicant so that the treating solutions contained less pentachlorophenol per unit volume at the end of the treating period than they did at the beginning. Whether the reduction in toxicant concentration was significant is not known.[1]

ABSORPTION OF SOLUTION

Tables 9 to 11 and Figs. 12 to 17 show the absorptions of solution in pounds per cubic foot[2] for various treatments.

The average absorption for all blocks was 6.31 pounds, ranging from 4.74 pounds for blocks treated at 20-percent moisture in a 40° F. solution to 11.30 pounds for those treated at 28-percent moisture in the 80° solution (Table 9).

Table 10 shows that the highest average absorption for the five levels of solution temperature (7.62 pounds) occurred at 80°, and the lowest (5.62 pounds) at 40° F. The absorptions at 5°, 10°, and 20° F. (6.12, 6.18, and 6.00 pounds, respectively) probably were not significantly different (Fig. 12).

The highest average absorption for the four levels of wood moisture content was 7.74 pounds for blocks treated at 28 percent (Table 10). The lowest absorption occurred at 20-percent moisture content. Thus absorption increased from 6.06 pounds to 6.43 pounds as moisture content increased from 7 to 13 percent, fell to 5.06 pounds at 20-percent level, and finally rose to 7.74 pounds for wood treated at 28-percent moisture (Fig. 13). The same trends are shown for continuous and intermittent treatments (Fig. 16) and for both solutions (Fig. 17).

The average absorption for the continuous treatment was 6.62 pounds, and the average for intermittent treatment was 6.00 pounds (Table 11).

[1] No measurement was made of the toxicant content of the treating solutions at the end of the tests. By the time the liquid interchange tests were completed and the data analyzed, approximately seven months had elapsed. During this time the treating solutions were exposed to a range of relative humidities which made it inadvisable to make any comparisons.

[2] Absorptions in terms of pounds of solution per cubic foot of wood will be given without further reference to the common denominator.

Table 9. — Absorption of Solution for Various Treatments
of Basswood Test Blocks

Solution temperature (° F.)	Wood moisture content (percent)	Average solution absorption (lb. per cu. ft.)				
		Continuous		Intermittent		
		Diacetone alcohol (light) solution	Hexylene glycol (heavy) solution	Diacetone alcohol solution	Hexylene glycol solution	Average
5	7	7.80	6.37	6.06	4.99	6.31
	13	8.55	5.43	6.06	5.81	6.43
	20	5.68	4.62	5.49	4.12	4.93
	28	7.80	6.80	7.30	5.68	6.87
10	7	7.43	6.06	6.56	5.37	6.31
	13	8.05	5.43	6.56	5.68	6.43
	20	6.06	4.81	5.68	4.81	5.31
	28	8.30	6.43	6.99	5.62	6.80
20	7	7.05	5.56	5.87	5.49	6.00
	13	7.68	5.24	6.74	5.81	6.37
	20	6.06	4.49	4.99	4.06	4.87
	28	8.05	6.00	6.93	6.31	6.80
40	7	6.93	4.49	6.31	4.49	5.49
	13	6.06	4.74	5.93	4.99	5.37
	20	5.37	4.18	5.74	3.68	4.74
	28	7.24	7.37	7.30	5.81	6.93
80	7	7.87	5.43	6.37	5.37	6.24
	13	8.55	7.43	9.36	5.62	7.68
	20	6.31	4.31	5.87	4.87	5.31
	28	12.55	12.36	11.30	9.24	11.30
Average..........................		7.43	5.87	6.62	5.37	6.31

An analysis of variance was used to determine whether absorption differences were significant (Table 12).

Statistical analysis of absorption data. The analysis of variance was applied to data in terms of grams of liquid absorbed per cubic centimeter of wood, avoiding conversion of individual measurements to pound-per-cubic-foot units, and appears in Table 12.

A suitable error term was not available for testing the wood-moisture variable, so a variance ratio is not shown in Table 12 for this source of variation.

Table 10. — Absorption of Solution by Test Blocks Treated at Different
Moisture Contents in Solutions of Different Temperatures

Wood moisture content (percent)	Absorption of solution (lb. per cu. ft.)					
	Solution temperature (° F.)					Average
	5	10	20	40	80	
7.....................	6.31	6.31	6.00	5.49	6.24	6.06
13....................	6.43	6.43	6.37	5.37	7.68	6.43
20....................	4.93	5.31	4.87	4.74	5.31	5.06
28....................	6.87	6.80	6.80	6.93	11.30	7.74
Average...............	6.12	6.18	6.00	5.62	7.62	6.31

Solution temperature had a variance ratio of 2.8, not quite large enough to be significant at the 0.05 level, and thus did not have a statistically significant effect on absorption. Although this result does not agree with those obtained by other investigators (6, 23, 24), the effect of temperature on solution viscosity and absorption is not likely to be as important within the range of temperatures tested (5° to 80° F.) as it is at temperatures above 100° F., the level studied by those investigators.

Effect of solution temperature and wood moisture. Although a suitable error term was not available for testing the TM interaction in the analysis of variance (Table 12), it is worth while to consider the possibilities of what appears to be an interacting effect of these two variables on solution absorption (Figs. 12 and 13 and Table 10).

If these two variables do have an interacting effect on the absorption of solution, it means that the amount of preservative solution absorbed as a function of temperature was not the same for each level of wood moisture (and vice versa). It also means that the two variables
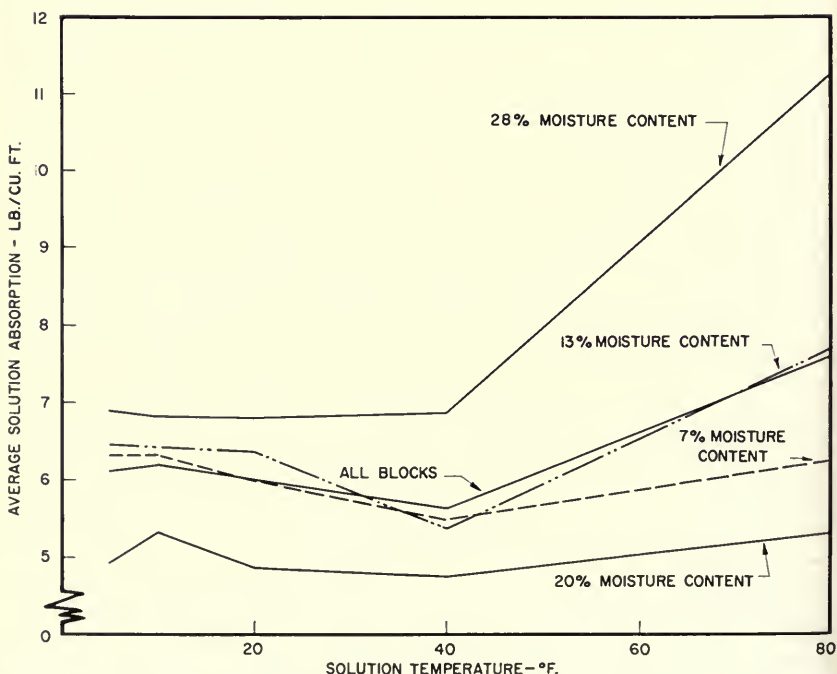
Effect of solution temperature on the absorption of solution by test blocks treated at different moisture contents.   (Fig. 12)
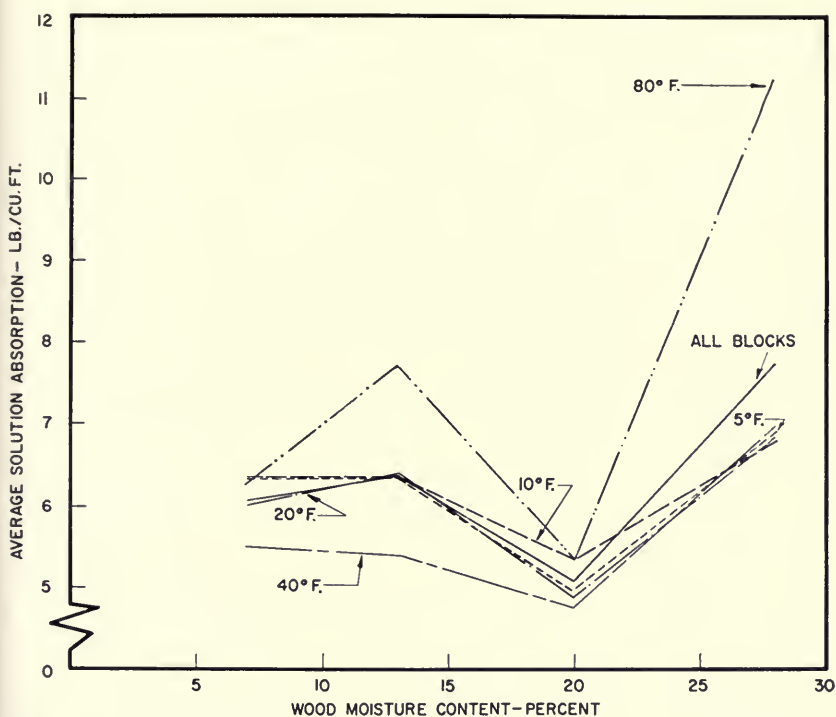
Effect of wood moisture content on absorption of solution by test blocks
treated in solutions of different temperatures.   (Fig. 13)

were interdependent in their effects on absorption, and their average
independent effects on solution absorption become relatively meaning-
less and unimportant. Such a relationship thus precludes the practice
of discussing treating results in terms of adjusting only the tempera-
ture of the treating solution or the moisture content of the wood.

The results show, for example, that there was no significant change
in the amount of solution absorbed by blocks treated at 28-percent
moisture content and in solutions at temperatures of 40° F. or cooler
(Fig. 12). Absorptions for blocks treated at 13-percent moisture, how-
ever, were about the same for solution temperatures up to 20°, but at
40° the absorption of solution by the 13-percent blocks was the lowest
recorded for all treatments at that level of moisture. The greatest
absorption for blocks treated at 13-percent moisture occurred at the
80° F. solution temperature.

According to Stamm (31, 32), the movement of an aqueous or non-
aqueous treating solution into softwoods treated by nonpressure meth-

ods involves a combination of capillary rise and liquid flow or, if a
concentration gradient exists, diffusion. The movement of liquids
through basswood may not be more complicated than the liquid flow
through softwoods, where practically the total resistance to flow exists
in the pit-membrane pores. The vessels and simple pits and perforation
plates of basswood probably do not inhibit liquid flow any more than
the corresponding anatomical feature of softwoods. For example, the
diameters of the pit pores in basswood range from 5 to 8 microns (5),
apparently about the same size of pit pores as in softwoods (20, 22, 31).
It is likely that the openings in the pit membranes of basswood also are
similar in size to those found in softwoods.

Hawley (12) concluded that the effect of the viscosity of a liquid
on its movement into wood was about what one would expect from the
known facts of wood structure and the laws governing the flow of
liquids into capillary tubes. The viscosities of the two treating solutions
are curvilinear functions of temperature; thus as solution temperature
was increased, the viscosities of the solutions were reduced. One might
conclude, as other investigators have (2, 23, 24), that as a consequence
of increased fluidity, the treating solutions penetrated the wood more
readily and solution absorption increased. If absorption differences re-
sulted *only* from a change in solution viscosity, the absorption curves in
Fig. 12 would rise at a curvilinear rate corresponding roughly to the
changes in solution viscosity. The lines show, however, that factors in
addition to changes in solution viscosity must have influenced absorption.

If we assume that all blocks were below the fiber-saturation point
(they apparently were), then the cell lumina contained no free water
and the treating solutions were free to move through the cell cavities by
capillarity. Thus moisture in the form of free water in the lumina had
no effect on the treating results.

Since oil-borne preservatives ordinarily move from one wood cell to
another through the lumina and pits in the cell walls, any factor affect-
ing the size of the cell lumina or the diameter of the pits and pit-
membrane pores affects liquid flow and ultimately the amount of liquid
absorbed by the wood. The size of the pit apertures increases as the
wood moisture content increases up to the fiber-saturation point (33).
If the pit membranes, which are of lignin, have a hygroscopicity similar
to that of wood, then the pit-membrane capillaries may also increase in
size with an increase in moisture content. Thus it appears that the
absorption of solution should have increased with increased moisture
content; however, because this reasoning does not account for the

fluctuating absorption shown by the line for all blocks in Fig. 13, we must seek another explanation.

Erickson *et al.* (10) concluded from their study of the flow of liquids through wood that there were other factors which might have greatly affected the permeability of a given wood to a given liquid. They found, for example, that benzene, a nonpolar compound, had little or no affinity for the polar wood substance and hence was more free to flow through the capillaries. Both of the solvents used in this investigation were polar compounds. Whether differences in polarity influenced absorption is unknown, but polarity in itself is believed to have an insignificant effect on absorption variation.

Absorption curves show that some inhibiting influence caused absorption to fall at 20-percent moisture content (Figs. 13, 16, 17). In measuring the air-pressure drops occurring through thin sections of wood exposed to air of different relative humidities, Stamm (31) found that the square root of the pressure-drop ratio increased linearly with a decrease in moisture content of the wood below 20 percent. Deviations in the linear regression at the 20-percent point were explained by the condensation of moisture in the pit-membrane pores which apparently took place when the relative humidity reached 90 percent. Wood attains a 20-percent equilibrium moisture content at such a relative humidity. The condensation of moisture in the pit-membrane pores apparently plugged them, thus eliminating them as a source of capillary flow. Therefore it is possible that an increase in moisture content of the basswood from 7 to 13 percent caused the pit capillaries to expand and the absorption of solution to rise; but in the blocks treated at 20-percent moisture content, moisture condensed in the pit-membrane pores and restricted the flow of solution through them, thereby causing the drop in the absorption curve. At 28-percent moisture content, however, the diameter of the pit capillaries increased, the inhibiting effect of the condensed moisture was overcome, and the relatively unrestricted flow of solution resulted in increased absorption. The relative positions of the curves in Fig. 12 also support the hypothesis that moisture condensation in the pit-membrane pores reduced liquid absorption.

The hypothesis is reasonable as long as the liquid moves into the wood by capillarity. However, Tarkow[1] points out that ". . . capillarity will generally occur only through the first, or perhaps the second, tier of cells," or until it is stopped by a constriction such as a pit-

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[1] Comment contained in letter to author by Dr. Harold Tarkow, U. S. Forest Products Laboratory, dated July 3, 1956.

membrane pore. Then hydrostatic pressure is required to overcome the resistance of the constrictions. The magnitude of the pressure in dynes per square centimeter can be determined by:

$$P = \frac{2\gamma}{r}$$

where γ is the surface tension of the liquid in dynes per centimeter, and r is the radius of the capillary. If the surface tension is 25 dynes per centimeter (hexylene glycol is about 33 and that for diacetone alcohol probably is greater than 25), and if the average radius of the pit-membrane pores is 3 microns $(3 \times 10^{-4}$ centimeter), the hydrostatic pressure would be about 16.7×10^4 dynes per square centimeter, or 2.5 pounds per square inch, a considerably greater pressure than that actually exerted by the 9-inch head of liquid in the treating tanks. Thus it seems reasonable that capillarity alone did not account for total absorption, but that the treating solutions moved into the wood as a result of a combination of capillarity rise and diffusion.

Figs. 12 to 15 show that absorption often was lowest when blocks were treated in 40° F. solution. If the condensation of moisture mentioned earlier took place at 40°, then the strongest inhibiting effect should be shown for blocks treated at 20-percent moisture content in the 40° solution. Table 9 shows that the absorption for blocks treated under such conditions was 4.74 pounds, the lowest for all treatments.
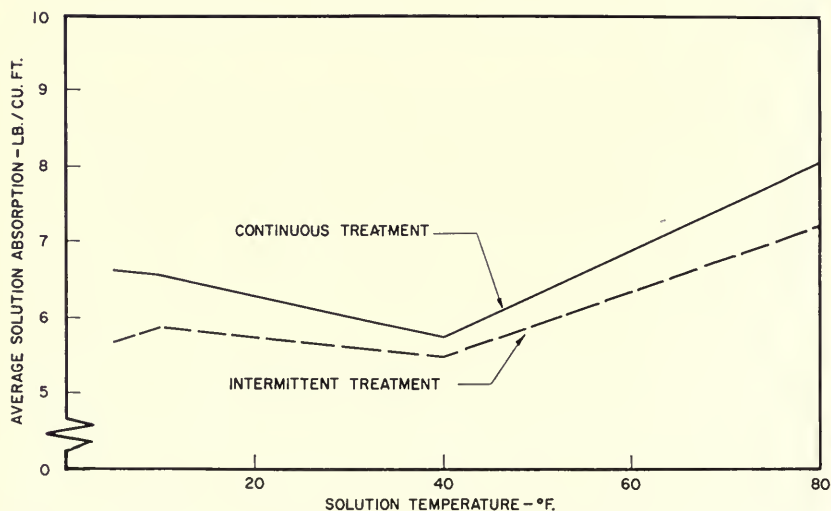
Effect of solution temperature on absorption of solution for two methods of treatment.   (Fig. 14)
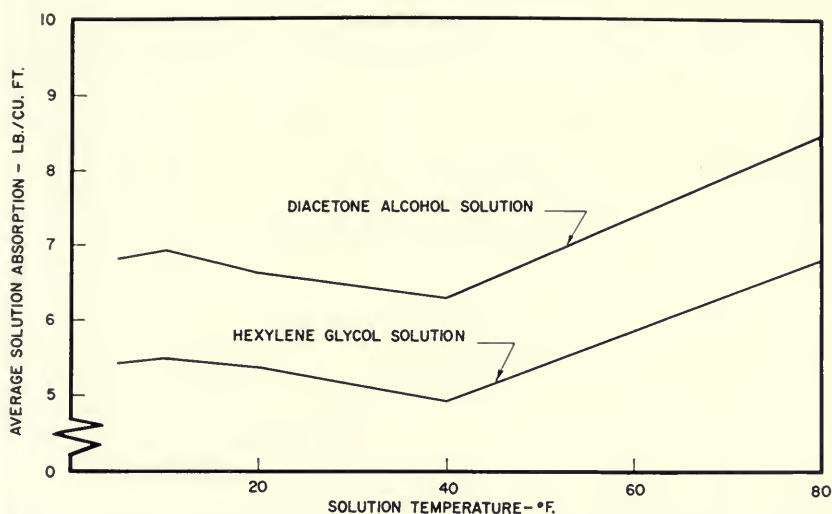
Effect of solution temperature on absorption of two solutions of penta-
chlorophenol.   (Fig. 15)

This explanation, however, becomes questionable when one observes in
Fig. 13 and Table 9 that absorptions of 40° F. solution also were low
at 13-percent and 7-percent moisture contents. It is doubtful that low
absorptions at these moisture contents resulted from the condensation
of moisture in the pit-membrane pores. Perhaps the cell-wall capil-
laries in blocks treated at 7 percent and 13 percent were somewhat
smaller than those in the 20-percent blocks and diffusion of liquid into
the cell walls was restricted.

Undoubtedly absorption was affected by a number of unknown
factors, as well as by factors which were known but uncontrolled. For
example, the effects of the crystallization of moisture in the cell-wall
capillaries of blocks treated at temperatures below freezing, the change

Table 11. — Absorption of Solution by Test Blocks Treated
in Two Solutions by Two Different Methods

Type of treatment	Absorption of solution (lb. per cu. ft.)		
	Diacetone alcohol (light) solution	Hexylene glycol (heavy) solution	Average
Continuous..........................	7.43	5.87	6.62
Intermittent..........................	6.62	5.37	6.00
Average..............................	7.05	5.62	6.31
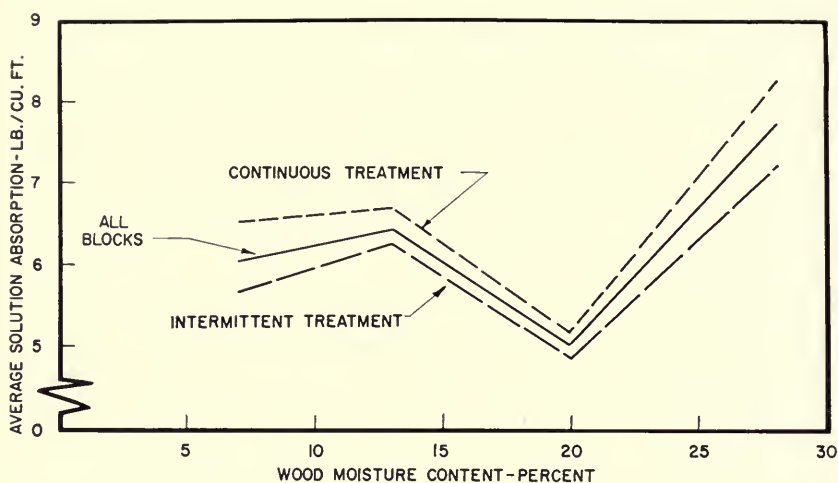
Effect of wood moisture content on absorption of solution for two types of
treatment.   (Fig. 16)

Table 12. — Analysis of Variance for Solution Absorption

| Source of variation	Degrees of freedom	Mean square	Variance ratio (F)
Wood moisture content (M)	3	.032118	(a)
Solution temperature (T)	4	.012143	2.8 NS[b]
TM (error)	12	.004256	. . . . . .
Type of treatment (D)	1	.010598	32.7***[c]
Solvent type (S)	1	.052969	163.5***
SD	1	.000569	1.8 NS
MS	3	.000319	1.0 NS
MD	3	.000788	2.4 NS
TS	4	.000125	.4 NS
TD	4	.000390	1.2 NS
MSD	3	.000519	1.6 NS
TSD	4	.000676	2.1 NS
MTD	12	.000284	.9 NS
MTS	12	.000210	.6 NS
MSTD	12	.000542	1.7 NS
Error	320	.000324	
Total	399		

[a] A suitable error term not available for test.
[b] NS, not significant at .05 level.
[c] *** Significant at .001 level.

in liquid viscosity caused by the addition of pentachlorophenol to the solvents, and the temperature differentials resulting from treating warm blocks in cold solutions (and vice versa) were not measured. The effects of such influences on solution absorption were beyond the scope of this study.

Effect of type of treatment. Table 11 and Figs. 14 and 16 show that continuous soaking was superior to intermittent treatment. The average solution absorption by blocks continuously treated was 6.62 pounds, whereas the average absorption for the intermittent type of treatment was 6.00 pounds. The difference is small, but statistically highly significant (Table 12). Since the head of liquid in each treating tank was approximately the same, hydrostatic pressure was constant as long as the blocks were completely submerged. The absorption differences are attributed primarily to difference in treating time. Although both groups were treated under the same conditions for 12 hours, the blocks treated intermittently were completely submerged in the treating solutions only 29 percent of the time.

Effect of type of solvent. Table 11 shows that the average absorption of the diacetone alcohol solution was 7.05 pounds and that for the hexylene glycol solution was only 5.62 pounds. Figs. 15 and 17 show that the absorption of the lighter or less viscous solution was greater at each level of moisture content and solution temperature. Table 12 shows that the difference in absorption was significant at the 0.001 level
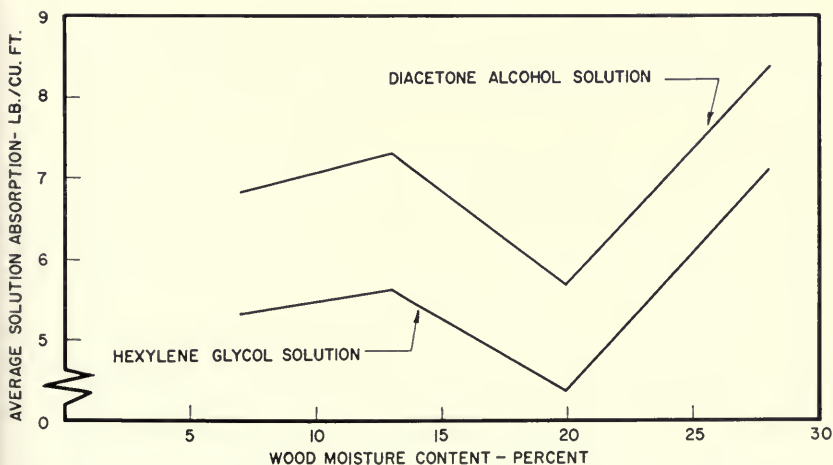
Effect of wood moisture content on absorption of two solutions of penta-
chlorophenol.   (Fig. 17)

Table 13. — Absorption of Solution by Test Blocks Treated
in Solutions of Different Temperatures by Two Methods

Type of treatment	Absorption of solution (lb. per cu. ft.)					
	Solution temperature (° F.)					Average
	5	10	20	40	80	
Continuous............	6.62	6.56	6.24	5.74	8.05	6.62
Intermittent...........	5.68	5.87	5.74	5.49	7.24	6.00
Average...............	6.12	6.18	6.00	5.62	7.62	6.31

of probability. Diacetone alcohol has a viscosity of 15.3 centistokes at
5° F. and hexylene glycol has a viscosity of 700.0 centistokes at approx-
imately 10° F. (Table 2). Thus the difference in absorption is at-
tributed to differences in viscosities. The "lighter" solution penetrated
the blocks better.

It is believed that the treatment in the frothy hexylene glycol had
no significant effect on absorption. The absorption values shown in
Table 13 for intermittent treatment are about 80 percent of those for
continuous treatment at all levels of solution temperature. If treatment
in the frothy solutions, which occurred at solution temperatures of 40°
and less, had had a significant effect on absorption, the effect would
have been shown by the above-mentioned comparison of absorption
values.

DISTRIBUTION OF PENTACHLOROPHENOL

Tables 14 to 18 show the gross[1] concentration of pentachlorophenol
in percent in the sample blocks. The average concentration was 0.696
percent, ranging from 0.507 percent for blocks treated at 7-percent

[1] No corrections have been made in the data to show net concentrations or
to convert the data to pentachlorophenol equivalencies, because the amount of
chlorides in the basswood and the reagents and the specific gravity of the bass-
wood were considered as constants in the analyses of the results. For those
who wish to make the conversions, however, the correction for chlorides in the
basswood and reagents was 0.055 percent, the average of seven analyses. The
average specific gravity of the basswood was 0.48, based upon oven-dry weights
and oven-dry volumes of three samples.

Some of the percentages in the tables exceed 2.0. Although the AWPA
standard method of analysis (8) indicates that the method ". . . is suitable for
analysis (of wood samples containing) up to 2.0 percent pentachlorophenol . . ."
the technique can be used as long as there is enough lime in the crucible to
capture all the chlorides produced. When the wood sample is large enough to
cause cavities or cracks to develop in the lime bed, the chlorides escape during
the ignition period, and the results give percentages that are low. Such voids
and cracks did not develop during the analysis of the test samples.

Table 14. — Concentration of Pentachlorophenol at Three Locations in Test Blocks Treated Under Different Conditions
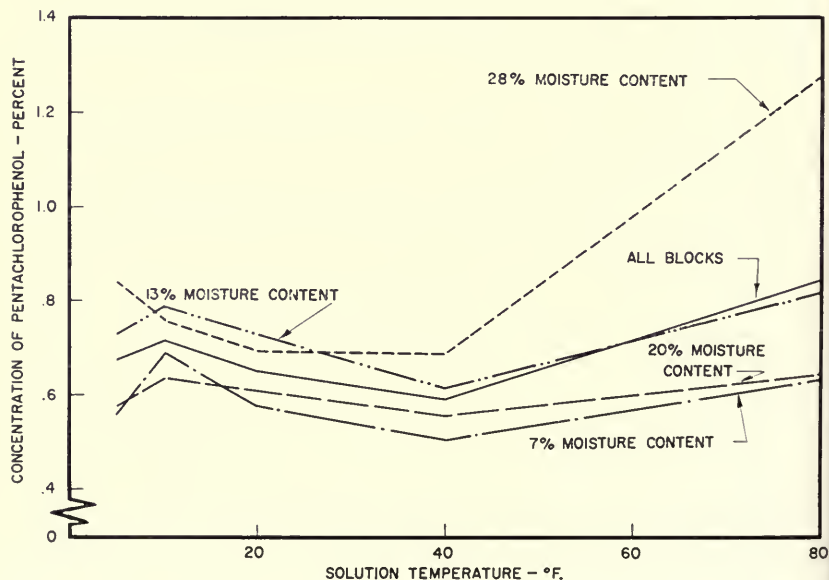
Solution temperature (°F.)	Wood moisture content (percent)	Continuous treatment						Intermittent treatment						Average
		Diacetone alcohol solution			Hexylene glycol solution			Diacetone alcohol solution			Hexylene glycol solution			
		Shell A	Shell B	Core	Shell A	Shell B	Core	Shell A	Shell B	Core	Shell A	Shell B	Core	
5	7	1.304	.503	.214	1.138	.322	.111	1.313	.402	.148	1.089	.164	.050	.563
	13	1.893	.777	.178	1.462	.241	.131	1.692	.434	.226	1.290	.346	.104	.731
	20	1.105	.147	.106	1.164	.252	.150	1.517	.361	.109	1.608	.347	.075	.578
	28	1.954	.633	.535	1.575	.537	.105	2.150	.670	.043	1.432	.314	.080	.836
10	7	1.569	.588	.138	1.368	.273	.082	2.166	.483	.164	1.190	.188	.072	.690
	13	2.356	.793	.123	1.296	.319	.107	2.129	.443	.125	1.295	.369	.122	.790
	20	1.835	.306	.149	1.355	.147	.108	1.647	.324	.082	1.351	.254	.086	.637
	28	1.903	.862	.141	1.409	.370	.169	1.807	.464	.172	1.315	.341	.152	.759
20	7	1.597	.453	.126	1.108	.235	.110	1.338	.319	.097	1.200	.210	.105	.575
	13	1.752	.561	.124	1.318	.312	.123	1.912	.660	.169	1.271	.347	.127	.723
	20	1.784	.299	.070	1.291	.241	.074	1.567	.333	.084	1.225	.242	.074	.607
	28	1.648	.372	.130	1.560	.251	.143	2.007	.350	.084	1.328	.343	.105	.693
40	7	1.485	.400	.155	.960	.103	.098	1.488	.374	.134	.672	.125	.095	.507
	13	1.547	.262	.161	1.396	.277	.108	1.429	.425	.151	1.265	.224	.140	.615
	20	1.555	.229	.085	1.092	.214	.083	1.528	.411	.095	1.130	.178	.045	.554
	28	1.626	.343	.073	1.675	.422	.067	1.921	.255	.108	1.357	.331	.078	.688
80	7	1.625	.562	.258	1.109	.627	.311	1.409	.295	.088	1.089	.147	.071	.633
	13	2.100	.597	.097	1.633	.614	.093	2.064	.657	.168	1.449	.314	.034	.818
	20	1.693	.476	.110	1.125	.244	.060	1.919	.283	.066	1.343	.327	.056	.642
	28	2.267	1.087	.810	1.755	1.319	.743	2.319	1.732	.454	1.820	.650	.324	1.273
Average		1.729	.512	.189	1.339	.366	.149	1.766	.484	.138	1.286	.288	.100	.696

Table 15. — Concentration of Pentachlorophenol at Three Depths
in Test Blocks Treated at Four Levels of Wood Moisture

Wood moisture content (percent)	Percent of pentachlorophenol			
	Shell A	Shell B	Core	Average
7	1.311	.339	.131	.594
13	1.627	.449	.131	.736
20	1.442	.281	.088	.604
28	1.741	.582	.226	.850
Average	1.530	.413	.144	.696

moisture in a 40-degree solution to 1.273 percent for those treated at
28-percent mositure content in the 80-degree solution (Table 14).

The distribution of toxicant, also referred to as "penetration," was
about what one would expect with regard to the effect of depth of
sample on concentration. The highest average concentration of penta-
chlorophenol (1.530 percent) was found in Shell A, the outermost
layer (Table 15). Somewhat less toxicant, 0.413 percent, was found
in Shell B, the second deepest sample tested, and the core sample
contained the least amount, 0.144 percent. These results suggest that

Effect of solution temperature on concentration of pentachlorophenol in
test blocks treated at different moisture contents.   (Fig. 18)

Table 16. — Concentration of Pentachlorophenol in Test Blocks
Treated at Different Moisture Contents in Solutions
of Different Temperatures

Wood moisture content (percent)	Percent of pentachlorophenol					
	Solution temperature (° F.)					Average
	5	10	20	40	80	
7	.563	.690	.575	.507	.633	.594
13	.731	.790	.723	.615	.818	.736
20	.578	.637	.607	.544	.642	.604
28	.836	.759	.693	.688	1.273	.850
Average	.677	.719	.650	.591	.842	.696

a toxicant diffusion gradient existed in the blocks. It is believed, how-
ever, that the differences in amount of toxicant were due to the pres-
ence of greater amounts of solution at each successive depth in the
blocks rather than to a concentration gradient.

The following discussion of the effects of solution temperature,
wood moisture content, type of treatment, and type of solvent on the
distribution of pentachlorophenol will be brief; a more comprehensive
discussion of the results is given later.

Table 16 and Fig. 18 show that the average concentration of penta-
chlorophenol increased from 0.677 percent to 0.719 percent as solution
temperature increased from 5° to 10° F., then fell to a minimum of
0.591 percent for the 40° treatment. The maximum concentration,
0.842 percent, was found in blocks treated in the 80° solution.

Table 16 and Fig. 19 show the pentachlorophenol concentrations
found in blocks treated at four levels of wood moisture. The lowest
concentration, 0.594 percent, was found in blocks treated at 7-percent
moisture. The toxicant concentration rose to 0.736 percent when treat-
ment was made at 13 percent, but fell to 0.604 percent, about the same
level as that found in blocks treated at the 7-percent level, when treat-
ment was made at 20-percent moisture content. The greatest amount
of toxicant, 0.850 percent, was found in the blocks treated at 28-percent
moisture.

Table 17 and Fig. 20 show that blocks treated by continuous soaking
contained more pentachlorophenol (0.714 percent) than those treated
by intermittent soaking (0.677 percent).

Table 18 and Fig. 20 show that the blocks treated in diacetone
alcohol solution contained more pentachlorophenol (0.803 percent)
than those treated in the hexylene glycol solution (0.588 percent).
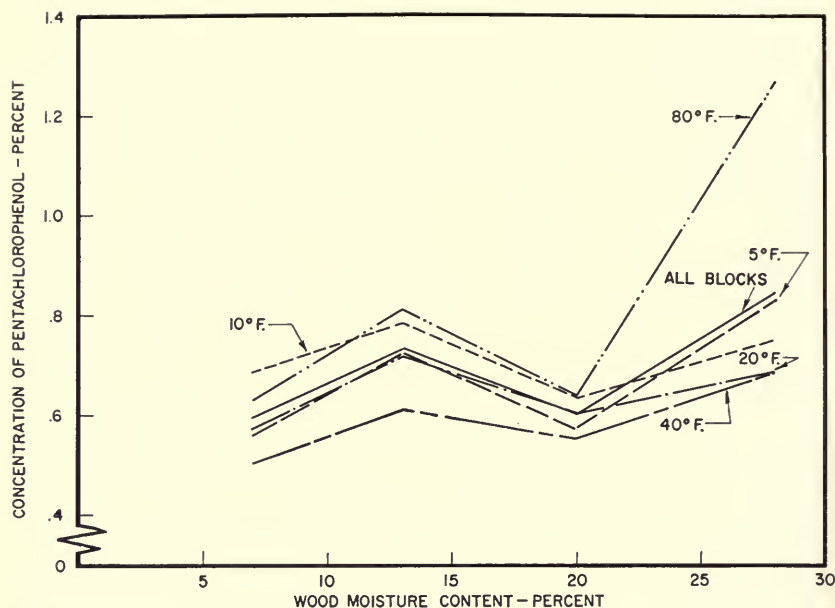
Effect of wood moisture content on concentration of pentachlorophenol in test blocks treated in solutions of different temperatures.   (Fig. 19)
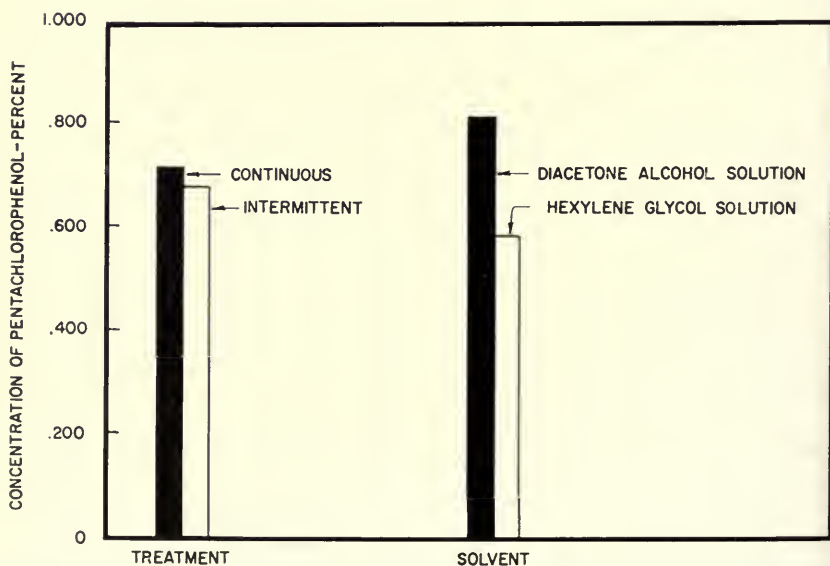
Effect of type of treatment and solvent on concentration of pentachloro-phenol in test blocks.   (Fig. 20)

Table 17. — Concentration of Pentachlorophenol at Three Depths in Test Blocks Treated by Continuous and Intermittent Soaking

Type of treatment	Percent of pentachlorophenol			
	Shell A	Shell B	Core	Average
Continuous...................	1.535	.439	.169	.714
Intermittent.................	1.526	.386	.119	.677
Average.....................	1.530	.413	.144	.696

A multivariate analysis was used to determine whether penetration differences were significant (Table 19).

Statistical analysis of penetration data. The multivariate analysis testing the significance of penetration results follows the method described by Rao (28), who refers to it as "analysis of dispersion." It is an extension of the univariate analysis of variance which was employed in the section on solution absorption.

A suitable error term was not available for testing the wood moisture variable, so a variance ratio is not shown in Table 19 for this source of variation.

Solution temperature was a nonsignificant source of variation. As mentioned earlier, this result does not substantiate results of earlier investigations of the effect of solution temperature on penetration (toxicant concentration in this study). However, the effect of temperature on solution viscosity and penetration into the wood is not likely to be as great within the range of solution temperatures tested (5° to 80° F.) as it is at higher temperatures, those above 100° F. for example.

Although a suitable error term was not available for testing the TM interaction — solution temperature and wood-moisture content — it is worth while to review what appears to be an interacting effect of these two variables on toxicant penetration (Figs. 18 and 19).

Effect of solution temperature and wood moisture. If we compare the concentration regressions in Figs. 18 and 19 with the absorption regressions in Figs. 12 and 13, we note a similarity in their patterns. It is reasonable to assume that the two sets of absorption and concentration curves should be similar, since a close relationship should exist between the amount of solution absorbed and the amount of toxicant found in the test blocks.

There is no question but that the treatment of blocks at 28-percent moisture content in the 80° F. solution was superior to all other treatments as far as quantity of toxicant in the wood is concerned.

The position of the line for treatments made at 20-percent moisture content does not show the inhibiting effects of moisture condensation in the pit capillaries on toxicant concentration, discussed in the analysis of absorption results, as convincingly as the absorption data do (Fig. 12). The positions of the 20-percent and 7-percent curves in Fig. 18 are so arranged, however, that if we allow for experimental error, we may conclude that there was no significant difference between these curves at most levels of solution temperature.

The family of curves in Fig. 19 shows about the same relationship between toxicant concentration and wood moisture content as is shown by the absorption data in Fig. 13. Here again, if we allow for experimental error, we may conclude that the cause-result relationship probably was the same as that described for absorption data; namely, that with an increase in moisture content, the diameters of the pit pores increased, the absorption of solution increased, and higher concentration of toxicant resulted. But in the blocks treated at 20-percent moisture content, moisture condensed in the pit capillaries and restricted the flow of solution through them, thereby causing a drop in the concentration of pentachlorophenol. At the 28-percent moisture level, however, the diameters of the pit and cell-wall capillaries were expanded to maximum size, the inhibiting effect of the condensed moisture was overcome, and the relatively unrestricted flow of solution resulted in maximum toxicant concentration.

An increase in solution temperature decreased solution viscosity, and toxicant concentration should have increased positively with temperature. However, Table 16 shows the depressing effect that the treatment of blocks in the 40° F. solution had on the absorption of solution and the concentration of pentachlorophenol in the wood. Thus the greatest inhibiting effect of solution temperature combined with wood-moisture content should have occurred when blocks were treated at 20-percent moisture content in the 40° F. solution. Table 16 shows that the concentration of pentachlorophenol for such a treatment was 0.544 percent. Although the concentration obtained by treating blocks at the same solution temperature but at 7-percent moisture content was lower (0.507 percent), a "t" test showed that the mean concentrations for the two levels of moisture were not significantly different.

There is also the possibility that the size of the cell-wall capillaries, which were expanded to maximum size at 28-percent moisture content, had an effect on pentachlorophenol concentration. This possibility has been discussed in the section concerning the interchange of moisture

Table 18. — Concentration of Pentachlorophenol at Three Depths in Test Blocks Treated in Solutions Containing Diacetone Alcohol or Hexylene Glycol

Solvent	Percent of pentachlorophenol			
	Shell A	Shell B	Core	Average
Diacetone alcohol.................	1.748	.498	.164	.803
Hexylene glycol.................	1.313	.327	.124	.588
Average.....................	1.530	.413	.144	.696

and solvent during treatment and in the discussion of solution absorption.

Effect of type of treatment. Table 17 and Fig. 20 show that continuous soaking resulted in a higher concentration of pentachlorophenol than intermittent treatment, 0.714 percent and 0.677 percent, respectively. Table 19 shows that the difference in chemical content was significant at the 0.01 level of probability. The difference is attributed to the greater length of time the blocks received treatment by the continuous method. The same relationship between the two types of treatment was found in the analysis of absorption data.

Effect of type of solvent. Table 18 and Fig. 20 show that the concentration of pentachlorophenol was 0.803 percent in blocks treated in the diacetone alcohol solution and 0.588 percent in the blocks treated in hexylene glycol solution. The difference in chemical content was signifi-

Table 19. — Multivariate Analysis[a] for Penetration Data

Source of variation	Degrees of freedom	Sum of products matrix						Level of significance[b]
		A^2	B^2	C^2	AB	AC	BC	
ood moisture content (M)...	3	2.200	1.059	0.203	1.345	0.473	0.433	[c]
ution temperature (T).....	4	0.862	1.024	0.180	0.803	0.281	0.418	NS
(error).................	12	0.660	1.226	0.512	0.499	0.220	0.733	..
pe of treatment (D).......	1	0.002	0.057	0.050	0.009	0.009	0.053	**
vent type (S).............	1	3.790	0.586	0.031	1.490	0.344	0.135	***
....	4	0.258	0.047	0.019	0.071	−0.039	0.002	NS
....	4	0.038	0.070	0.074	−0.022	−0.038	0.050	NS
....	3	0.075	0.068	0.005	0.067	0.013	0.015	NS
....	3	0.084	0.090	0.057	0.055	−0.020	0.026	NS
....	1	0.040	0.012	T^d	0.022	−0.001	T^d	NS
or.....................	43	1.262	0.981	0.186	0.289	0.061	0.030	
al.....................	79	9.272	5.221	1.316	3.377	1.303	1.896	

[a] Called "analysis of dispersion" by Rao (28).
[b] *** Significant at .0001 level.
    ** Significant at .01 level.
[c] A suitable error term not available for test.
[d] T, trace or less than 0.001.

cant at the 0.001 level of probability (Table 19). The same results were obtained in the analysis of absorption data, and they were attributed to the difference in viscosities of the treating solutions. The diacetone alcohol solution had the lower viscosity and was able to penetrate the blocks easier than the more viscous hexylene glycol. The greater concentration of pentachlorophenol probably was due to greater absorption of the lighter preservative solution.

Summary and Conclusions

The absorption of solution and the concentration of pentachlorophenol were determined following constant and intermittent cold-soak treatments of basswood blocks of different moisture contents in solutions of different viscosities. Solution temperatures ranging between 5° and 80° F. had a statistically nonsignificant effect on the amount of solution and toxicant absorbed by the wood. No suitable error term was available for testing the effect of wood moisture content on absorption of solution and toxicant. No interaction of variables tested was found statistically significant, although an explanation is presented which accounts for what appears to be an interacting effect of solution temperature and wood moisture content on solution absorption and toxicant concentration.

The minimum absorption occurred when the blocks were treated at 20-percent moisture content in a 40° F. solution, an intermediate level for both variables. The result was attributed to the inhibiting effects of moisture which condensed in the pit capillaries under the moisture-temperature conditions. Approximately the same result was obtained when toxicant concentration was measured for such a treatment, and the same conclusions were reached.

The highest absorption of solution and concentration of toxicant were found in blocks treated at 28-percent moisture content in an 80° F. solution. From these results it was concluded that increased solution temperature lowered viscosity and improved liquid flow, whereas an increase in wood moisture content to 28 percent caused the pit and cell-wall capillaries to expand to maximum diameter, allowing a maximum amount of solution to flow into the wood.

Continuous treatment was found to be superior to intermittent treatment during a 12-hour soaking period because of the shorter time the intermittently treated blocks were completely submerged in solution.

A low-viscosity solution of pentachlorophenol containing diacetone alcohol solvent was absorbed in greater quantities than a more viscous

solution containing hexylene glycol solvent. The concentration of pentachlorophenol also was greater in blocks treated in the diacetone alcohol solution. It was concluded that the greater absorption and toxicant concentration were due to the lower viscosity of the diacetone alcohol solution.

Dimensional changes in basswood wafers following a 12-hour con- tinuous treatment in the solvents showed that wafers shrank slightly radially and swelled in a tangential direction. The moisture content of the solvents increased during the treatment, even though the treatments were conducted in containers sealed to prevent exposure of the solvents to the atmosphere. It was concluded that an interchange of moisture in the cell walls of the wood and solvent took place. The radial shrink- age was attributed to crooking of the ray cells, whereas the tangential swelling was due to penetration of the cell-wall capillaries by the organic solvents. The interchange of moisture and solvent which took place in the cell-wall capillaries indicates the possibility of a moisture- treating solution interchange. Such an interchange probably affected the absorption measurements, making them conservative, and indicated that diffusion, as well as capillarity, caused liquid to move into the wood.

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